48549704

STUDY TITLE

ENFORCEMENT ANALYTICAL METHOD FOR DDVP PEST STRIP FORMULATIONS

DATA REQUIREMENTS

Guideline Series OPPTS 830.1800 Enforcement Analytical Method

AUTHOR

Harris Shen
Study Director

TESTING FACILITY

AMVAC Chemical Corporation
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AMVAC CHEMICAL CORPORATION STUDY NUMBER

MET08701

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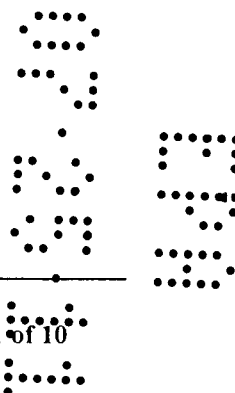## AUTHOR

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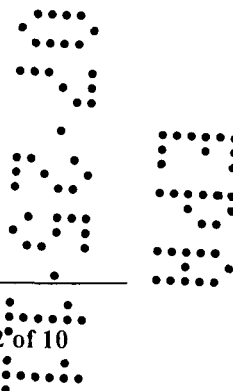
STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA, Section 10(d) (1) (A), (B), or (C).

Company: Amvac Chemical Corporation

Authorized Agent: Kaila Moran

Title: Regulatory Product Manager

Signature: _Kaila Moran_   Date: _7/19/2011_

STUDY GLP COMPLIANCE STATEMENT

The information presented herein was not conducted according to 40 CFR Part 160, GOOD LABORATORY PRACTICE STANDARDS (FIFRA), as promulgated in Federal Register, 54, No. 158, 34067-34704, 17 August 1989. There was no protocol or QA Unit review of the study.

Sponsor/Submitter: _Kaila Moran_ 7/19/2011

Kaila Moran
Regulatory Product Manager
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Management: _D Venter_ 7/19/2011

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Date

ENFORCEMENT ANALYTICAL METHOD FOR DDVP PEST STRIP FORMULATIONS

TABLE OF CONTENTS

METHOD OF ANALYTICAL AND METHOD VALIDATION

INTRODUCTION

Concentrations of the active ingredient, dichlorvos, in the test substance, Pest Strip, were determined by extracting with chloroform before GC analysis using flame ionisation detection (GC-FID).

REAGENTS AND SOLUTIONS

Materials
Chloroform, HPLC grade

PROCEDURES

Generation of Calibration Standard
Dichlorvos (DDVP) was accurately weighed (136.89 mg) into a 50 mL volumetric flasks and made to volume with chloroform. This solution was further diluted with chloroform to provide calibration standards at nominal concentrations ranging from 70 to 1600 µg/mL. These solutions were interspersed with the samples within the injection sequence.

Generation of QC Standards
Dichlorvos (DDVP) was accurately weighed (112.58 mg) into a 50 mL volumetric flasks and made to volume with chloroform to provide a QC sample at 2240.34 µg/mL . This solution was further diluted with chloroform to provide QC standards at 1120.17 and 168.03µg/mL. These solutions were analysed with the study samples within the injection sequence.

Analysis of Pest Strip Extracts
Extracts of Pest Strip in chloroform were analysed, in duplicate, without further dilution.

Conditions for GC Analysis with Flame Ionisation Detection

Instrumentation:	Varian 3300 GC with FID detector
Column#:	DB-5 30 m x 0.53 mm, 1.50 µm
Injection volume:	1 µL, split 1:10
Carrier gas:	Nitrogen
Make up gas:	Nitrogen
Head pressure:	5 psi
Column temperature:	100°C, hold for 1 min. Ramp at 20°C/minute to 240°C, hold for 1 min Ramp at 50°C/minute to 300°C, hold for 5 mins
Injection temperature#:	260°C
Detector temperature#:	300°C

Parameters marked # should not be modified. Minor adjustments to the remaining parameters may be required in order to fully optimise the system.

A typical chromatogram of a calibration standard containing dichlorvos is shown in Figure 3. A typical chromatogram of a QC standard is shown in Figure 4. The peak at a retention time of 5.12 minutes is due to dichlorvos.

Calculation of Results

All peak measurements and calculations were performed on Atlas, version 2000R2, using interspersed calibration standards, assuming a linear fit, with the formula below.

$$A \ (\mu g/mL) = \frac{\text{Peak Area - Constant}}{\text{1st Degree}} \ x \ \frac{UP3 \ x UP2}{UP1}$$

Where:-

A (µg/mL)	= Amount of dichlorvos
Peak Area	= Area of peak due to dichlorvos
Constant	= Y intercept on calibration graph
1st Degree	= Slope of calibration graph
UP1	= Sample amount
UP2	= Final volume (mL)
UP3	= Dilution
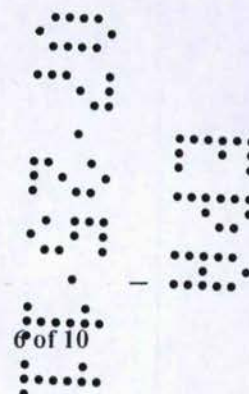
VALIDATION REPORT

This Analytical Procedure for dichlorvos in Pest Strip was validated using draft analytical procedure CLE PC 1708-052-02D. This validation was completed on 23 March 2006. The calibration curve obtained is shown in Figure 5, this correlation coefficient for this curve was 0.999.

The accuracy and repeatability (error in precision) were determined over 5 injections of QC samples prepared at nominal concentrations of 168, 1120 and 2240 μg/mL. The results are presented in the table below:

Precision and Accuracy Data for Dichlorvos		
Concentration (μg/mL)	Accuracy (%)	Error in precision (%)
168.03	93.57	4.879
1120.17	92.21	1.799
2240.34	92.45	3.808

Results obtained from Atlas run sequence Method validation 4

Accuracy = (mean of five determinations x 100 ÷ expected value)

Error in precision = (coefficient of variation, standard deviation x 100 ÷ mean of five determinations).

Figure 1
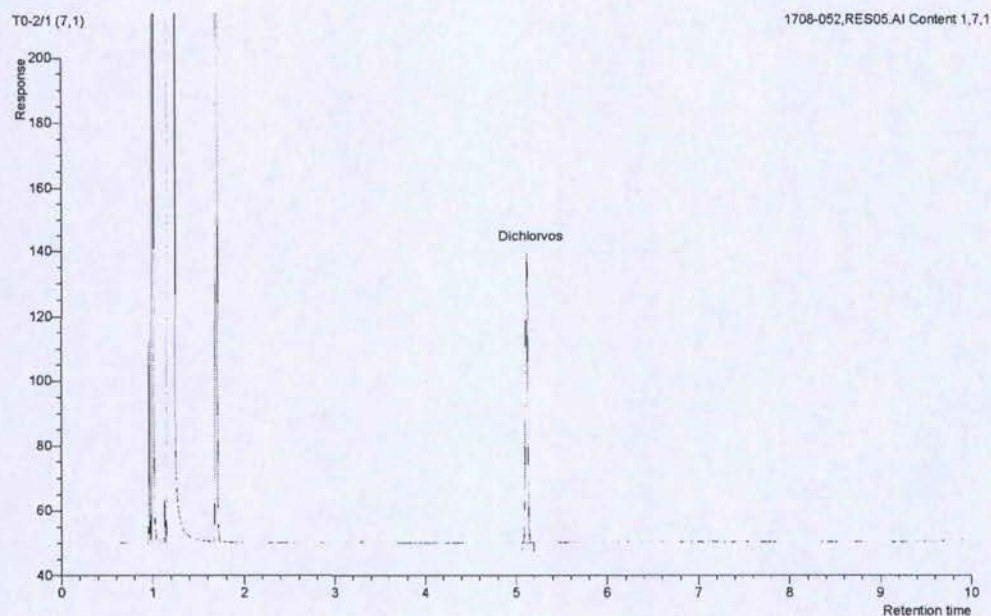## Example Chromatogram of an Extract from a Pest Strip Sample

T0-2/1 (7,1)

1708-052,RES05.AI Content 1,7,1

Dichlorvos

Figure 2
Example Chromatogram of an Extract from a Pest Strip Sample after the Elevated Temperature Storage Stability Study

ETS 1 1/2 (15,1)
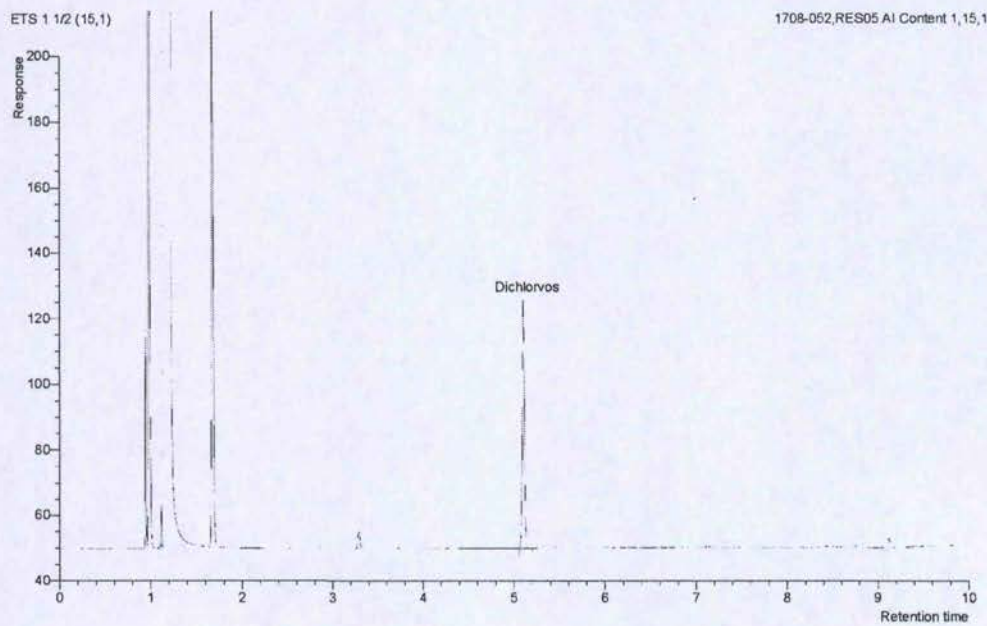
1708-052,RES05 AI Content 1,15,1

Dichlorvos

Figure 3
Example Chromatogram of a Calibration Standard

Cal 1600 (11,1)

1708-052,RES05 Validation 4,11,1

Response

Dichlorvos

200

180

160

140

120

100

80

60

40

0   1   2   3   4   5   6   7   8   9   10

Retention time
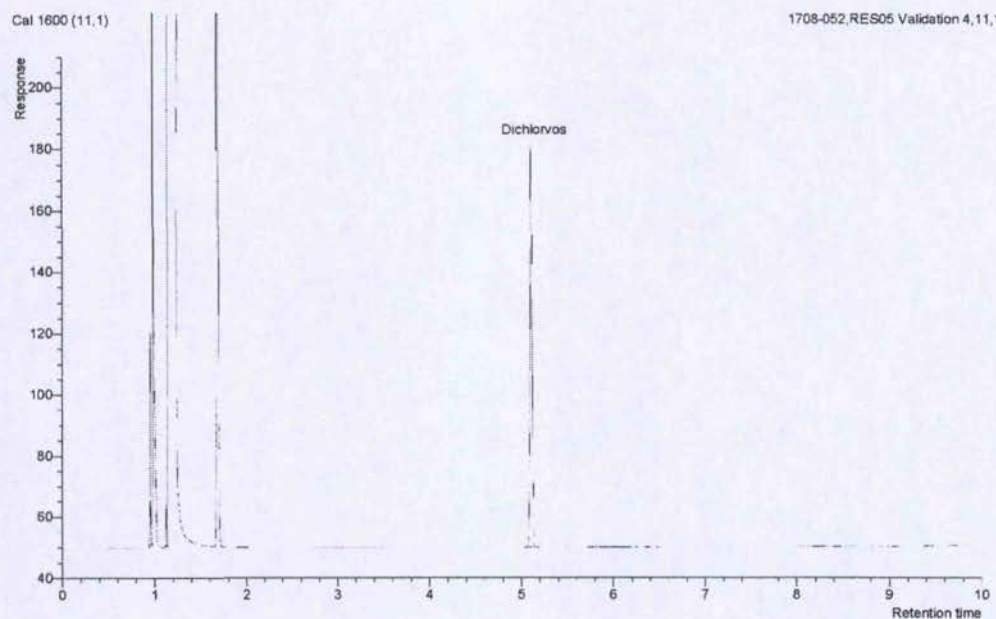
Figure 4
Example Chromatogram of a QC Standard

Mid QC (14,1)

1708-052,RES05 Validation 4,14,1

Response

Dichlorvos

200

180

160

140

120

100

80

60

40

0   1   2   3   4   5   6   7   8   9   10

Retention time

AMVAC Study No. MET08701
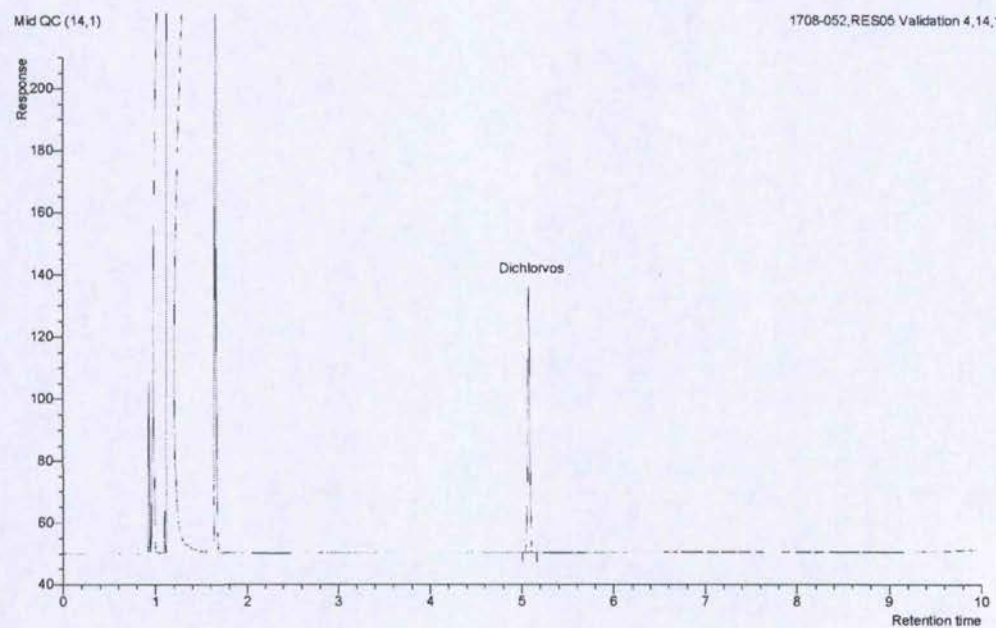
Figure 5
Method Validation Calibration Curve

Mid QC
RF Mode: Standards

Dichlorvos
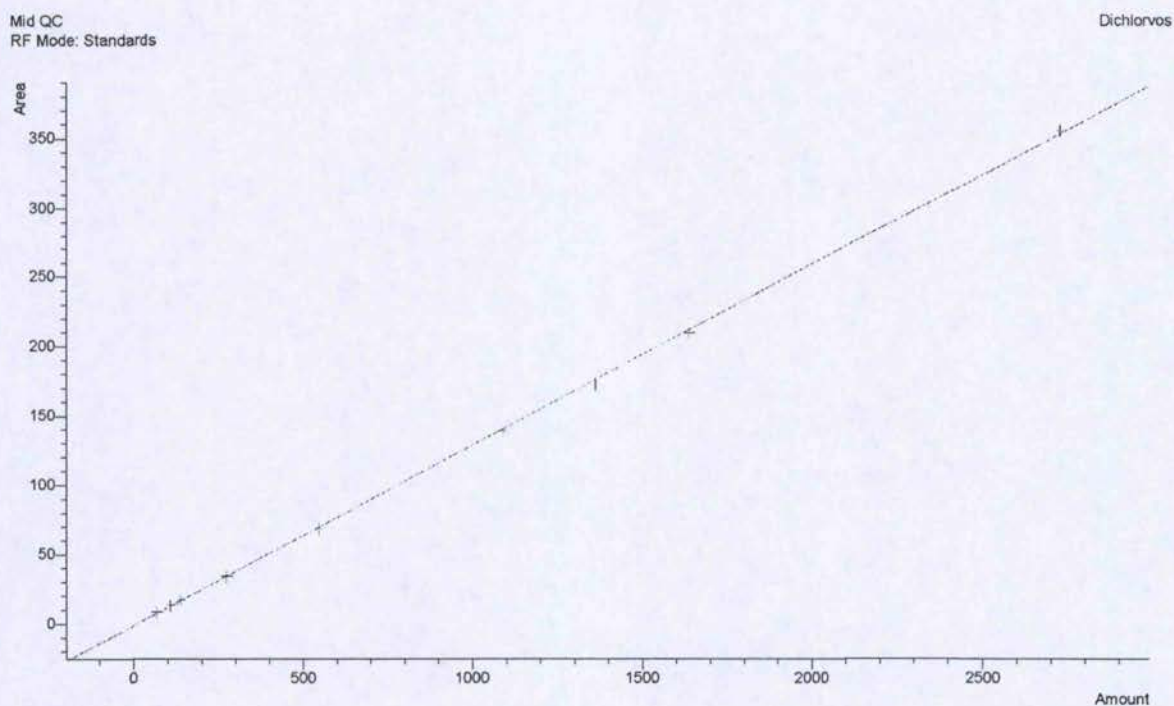
Calibration obtained from Atlas sequence 1708/052 Validation 4